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POROUS ALUMINA-BASED CERAMIC

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The possibility of producing strong porous ceramics based on aluminum oxide is shown. SiC with an activating additive and ammonium bicarbonate are used as a binder and a pore agent, respectively. The effect of the amount of the additives thus introduced and firing temperature on the porosity and strength of the material is studied.

Most ceramic filter elements used in industry contain a thin selective layer, a membrane deposited on a porous substrate which ensures the desired strength of the membrane. In service, filters are often subjected to the effect of increased pressure and mechanical loads which necessitates the use of high-strength materials in their production. However, the substrate should have high open porosity, permeability, and a uniform distribution of pores in size to reduce resistance to the inlet stream. We managed to develop an adequate material for the membrane base using alumina powder.

Starting material: GK alumina, average spherulite size 70 – 120 μm, hardening admixture: SiC powder with an activating additive [1].

This admixture substantially reduces the firing temperature of corundum articles via promotion of liquid-phase sintering.

The effect of the quantity of sintering admixture and firing temperature on the porosity and strength of the material was considered. A binder (3 – 5 wt.%) mixed with alumina and presses at 50 MPa was then fired at the temperatures listed in Table 1.

The porosity of the samples is 35 – 40%, and the bending strength is about 30 MPa. Firing shrinkage at the maximum firing temperature is 5 – 6%.

Introduction of decaying components that form active alumina is a powerful method of increasing porosity and hardening. Here we used aluminum hydroxide which is formed upon thermal decomposition

$$Al(OH)_3 \rightarrow \gamma\text{-}Al_2O_3 \rightarrow \alpha\text{-}Al_2O_3.$$

Losses of aluminum hydroxide upon calcination are about 35% [2]. Thus we expected an additional increase in porosity. The composition of the starting powders was as follows (%): 85 GK, 5 sintering admixture, 10 $Al(OH)_3$; 75 GK, 5 sintering admixture, 20 $Al(OH)_3$; and 65 GK, 5 sintering admixture, 30 $Al(OH)_3$.

The samples are pressed at 50 MPa and then fired at a temperature of 1350 – 1450°C. Proceeding from the data of petrographic analysis we established the phase composition of fired samples. Spherolite boundaries consisted of α-Al_2O_3. Fibrous clinoenstatite crystals (5 – 6 μm) are arranged along the spherolite boundaries. A fine-grained Al_2O_3 phase and

TABLE 1

Content	Sample properties			
	water absorption, %	open porosity, %	bending strength, MPa	linear shrinkage, %
Activating additive, %		*1300°C*		
3	33	62	10	0
5	31	58	15	2
		1350°C		
3	34	57	14	0
5	26	50	17	2
		1400°C		
3	21	43	19	3
5	17	37	24	5
		1450°C		
3	18	40	29	5
5	15	35	34	6
Alumina, %:		*1300°C*		
10	27	52	8	9
20	27	52	9	9
30	29	53	9	9
		1400°C		
10	25	49	21	13
20	26	50	18	11
30	26	49	19	11
		1450°C		
10	20	43	38	12
20	19	41	31	15
30	20	41	34	15

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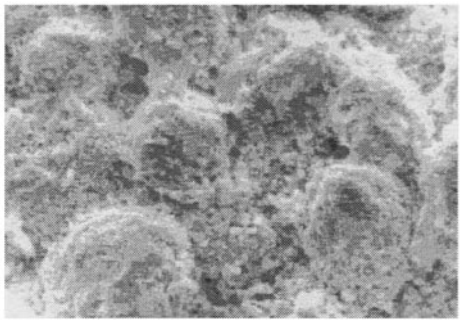

Fig. 1. Microstructure of porous ceramic (× 500).

transient $\gamma - \alpha$ modifications are present in accordance with the amount of aluminum hydroxide introduced. The size of these crystals is about 3 – 4 μm. The properties of the samples thus obtained are listed in Table 1.

Introduction of aluminum hydroxide ensures rather high porosity of the samples (41 – 43%) even at 15% linear shrinkage. Bending strength is 31 – 38 MPa depending on the content of aluminum hydroxide.

Aluminum hydroxide with a sintering additive exhibits high activity to sintering and even at a temperature of 1450°C sinters to a vitrified state: open porosity is no more than 1%, bending strength is 250 MPa, and shrinkage is 40%. However, introduction of aluminum hydroxide without sintering additive does not activate (promote) sintering at this temperature (Table 2). The composition (%) of the starting powders used for sample preparation was as follows: 90 GK, 10 $Al(OH)_3$; 80 GK, 20 $Al(OH)_3$; and 70 GK, 30 $Al(OH)_3$.

The bending strength of samples free of the sintering additive is rather low (2.5 – 5.0 MPa) at a porosity of 55 – 59%.

To evaluate the degree of strengthening of the samples after firing we used highly refined Al_2O_3 powder of Koral composition.

Introduction of additives fully decomposable upon firing results in the formation of open pores in the material and thus increases the porosity [3]. We examined a number of cellulating agents (both of organic and inorganic nature) and found that ammonium oxalate and ammonium bicarbonate appeared most acceptable.

In subsequent experiments we added ammonium bicarbonate (in excess of 100%) in amounts of 15, 35, and 50% to the starting powder (85 GK, 5 sintering additive, 10 Koral). The samples pressed at 50 MPa are fired at a temperature of 1450°C and kept for 2 h prior to determination of their properties (Table 3).

Using the most promising (for further investigations) material containing 15% of ammonium bicarbonate we pressed flask-shaped samples at the same pressure and then fired them at a lower temperature (1400°C) to increase the porosity of the article.

TABLE 2

Content of $Al(OH)_3$, %	Sample properties		
	water absorption, %	open porosity, %	bending strength, MPa
10	32	56	5
20	32	56	5
30	36	59	3

TABLE 3

Content of $(NH_4)_2CO_3$, %	Sample properties	
	open porosity, %	bending strength, MPa
15	28	74
35	47	11
50	59	7

Properties of the samples

Open porosity, %	42
Bending strength, MPa	45
Tensile strength, MPa	13
Pore size, μm:	
average	2
maximum	5
Average form factor of the pores	0.65

The microstructure of the samples is shown in Fig. 1.

The flasks were also tested for the efficiency of filtration (0.2 – 0.5 μm particles of a turbine oil, average size of particles 0.21 μm).[2]

Air consumption, cm^3/sec	104
Linear filtration rate, cm/sec	1
Filtration efficiency, %	97
Air resistance, Torr	30

Thus, addition of ammonium bicarbonate (as a pore forming agent) to the starting mixture is shown to increase the porosity of the material. In combination with a hardening additive ammonium bicarbonate makes it possible to get a high-strength porous material which can be successfully used in the production of filters and membrane bases.

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[2] Filtration indices were determined by A. V. Zagnit'ko of the Kurchatovskii Institute Scientific Research Center.